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ON THE METALLOGRAPHY OF ALUMINUM

by

Gunther Gonschior
and Erna Weiland

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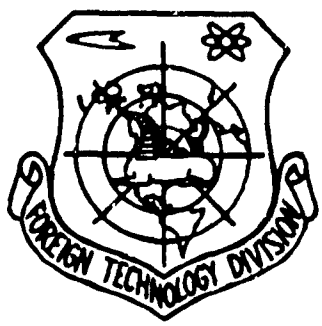

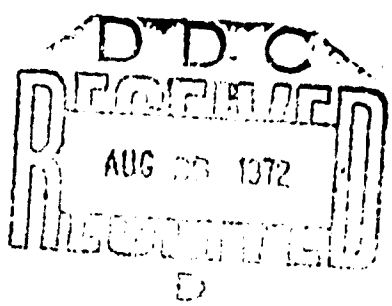

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A metallographic method was described in which dislocation etchings are obtained in minimum time and microstructural details can be obtained from samples corresponding to purities of Al 99.99 and Al 99.7. The steps involved in the technique are grinding, electrolytic polishing, dislocation etching, and anodic oxidation. Some examples obtained with the aid of this method were described. The method provides data on the presence of two phases, their configurations, and distribution; the density and distribution of dislocations on the basis of etched figures; the orientation of the crystals; and recovery and recrystallization. Grain-size determination is also possible. The method is suitable for AlMg3 and AlMgSi also. [AP0147044]

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ON THE METALLOGRAPHY OF ALUMINUM

Gunther Gonschior and Erna Weiland

REPORT FROM THE VEB MINING AND METALLURGICAL COMBINE, RESEARCH INSTITUTE FOR NONFERROUS METALS, FREIBERG

The steadily increasing requirements for the quality of aluminum and aluminum alloy products as used in the construction, packing and transportation industries inevitably requires more stringent control during their manufacture, beginning with the cast and extending to hot and cold shaping, following suitable heat treatment, etc., to the finished product. In this connection, the metallographic inspection even today still has an important place. With older methods of preparation the production of a perfect ground section is very time consuming. In addition, interesting information can be obtained on the structural details which cannot be obtained by this method. The problem thus also deals with mass production methods in preparing ground sections in connection with which the developed structure is to be able to give a great deal of information.

Schippers [1] describe a technique in which the granular structure of aluminum specimens can be made visible through anodic oxidation.

In the following article the application of a metallographic method is reported which has the purpose to obtain dislocation etchings in the shortest possible time and to give microstructure details of samples corresponding to purities of Al 99.99 and Al 99.71.

1. The Structural Development

The following procedural steps are to be carried out in which the given procedure must be maintained.

1.1. Grinding the specimens

All specimen shapes are suitable whether they are from cast pieces, from sheet bars, of wire or from foils. If the surface of the specimen is flat, if a photograph of the structure is not made, then it can be immediately electrolytically polished after the specimen is removed. Otherwise, wet grinding is the most practical method for surface smoothing.

1.2. Electrolytic polishing

In this step the superficial deformed layer is to be removed and the section is to be polished. The current direction of the electrolytes must be perpendicular to the previously mentioned polishing direction. The electrolytic polishing process should be stable. Basic requirements for this are a stable laminar electrolytic current and voltages between 50 to 60 V. If the temperature of the electrolyte is 20°C then these currents must increase the temperature of the specimen to approximately 100°C within 10 seconds. Since the temperature increase can lead to structural changes (and possibly even to an explosion in the case of alcoholic perchloric acid electrolytes!) the electrolyte was cooled by means of solid carbon dioxide to 0°C and the heat capacity of the sample holder was increased. In this way it was possible to hold a temperature of the probe below 10°C.

The Disa electropole of the Struers factory was electropolished. The voltage and time parameters must be determined by preliminary tests. Very good polishing results were obtained by using the Al-electrolyte of Arolda (perchloric acid density 1.6-8 mℓ; methyl alcohol 175 mℓ, glycerin 25 mℓ, methylglycol 100 mℓ) and the above described receptors for the Disa electropole.

1.3. The dislocation etching

Under certain conditions it is possible to localize the etching effect in such a way that the material that is removed is primarily in the immediate region of the defective point of the surface, particularly at the point at which the dislocation penetrates. Etching grooves then occur the forms of which correspond to the voltage field on the defective point. Rectangular or triangular etching patterns result in aluminum in the unshaped state, with their help a crystallographic orientation determination of the etched surfaces is possible.

In the investigations that were conducted an alcoholic $HCl-HNO_3$ solution is used. The etching process can be well observed due to the gas development that occurs. On very pure aluminum specimens displacement densities of up to approximately $10^8/cm^2$ were measured. The displacement density is very inhomogeneous. Figures 1a to c show examples of dislocation etching. The sharp edges of the etching figures are flattened out by the subsequent anodic oxidation due to the oxide film buildup. It is therefore recommended to take photographs of the etching figures prior to anodic oxidation.

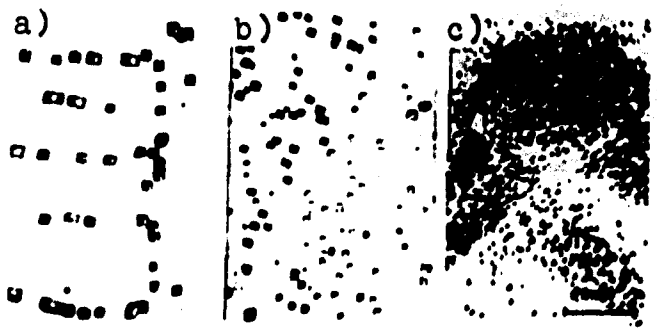

Fig. 1. Very pure aluminum. a) Chill cast iron: the arrangement of the etching grooves makes a specific relationship of the solidification direction noticeable; b) the different orientation of two grains is shown by small etching grooves (light and orientation effect); c) the specimen was rolled for approximately 40% at room temperature. The small etching groove density is very inhomogeneous.

1.4. Anodic oxidation

Anodic oxidation was carried out by Barker [2]. This method is suitable for pure aluminum as well as for aluminum alloys. A thin oxide layer grows on the grinding surface which makes the otherwise optical isotropic crystal surfaces of the aluminum appear to be anisotropic. The oxidation equipment consists of a direct current source of 60 V/5 A which can be controlled and of an electrolysis tub with a capacity of approximately [illegible]. The Schipper mounting and the cathodes are of very pure aluminum and are attached to a stand. The ground surface must be exactly opposite to the cathode. The electrolyte, according to Schipper, should consist of one part of fluoroboric acid and 20 parts of distilled water. As our test has shown, however, the concentration ratio of the electrolyte is variable over a large range, without influencing the results. The specimens that were used had a surface of approximately 3 cm^2. With one bath filling, 200 specimens could be anodized. The electrolyte did not display any aging effects within 14 days. The operating temperature is much more critical than the composition of the electrolyte. It should not be in excess of 20°C. In addition, in order to assure a good contact the specimen must be held

with the specimen holder so that the heat that is generated can be conducted away. Otherwise, the electrolyte boils on the specimen in spots. It is then impossible to anodize. The installation of a cooling coil and a stirring mechanism is recommended.

Anodization was carried out approximately from 1 to 2 minutes at from 20 to 40 V (specimen surface ~3 cm^2). The specimen was then washed with water and alcohol. If a bright yellow film is seen on the anodized surface during drying in warm air, then the oxidation treatment is sufficient. When observed in polarized light it was recommended to use a compensation filter. The Epityp was equipped with a very good polarization equipment.

2. Several Examples from the Investigations

The structure of a rolled sheet bar is shown in Fig. 2a which was rolled about 40% at a terminal rolling temperature of 420°C and quenched immediately in water. Recrystallization did not occur in connection with the hot reworking. The deformation bands were polygonal. When reheated for 1 h/500°C the structure recrystallized (Fig. 2b). Without the gray color of the grains (black-white picture), a fault with respect to the size of the individual grains, was not disregarded. In the inspection with polarized light, deformation bands are very easily seen, because the orientation differences can be seen in the color (Fig. 2c). In order to more accurately characterize the deformation bands, slip line patterns can be produced (Fig. 3) following the dislocation etching and prior to anodizing (e.g., by pressure). Disorientations, even the smallest kind, are made clear in this way. The width and density of these bands may be considered as a rough scale of the structural strength.

Fig. 2. Very pure aluminum. a) The specimen is rolled approximately 40% in connection with a final rolling temperature of 420°C. The grains are stretched and the deformation bands are polygonal; b) the specimen in Fig. 2a was heat treated for 1 h at 500°C whereupon the structure recrystallized; c) very pure aluminum which was rolled 60% at a terminal rolling temperature of 420°C. Formation bands were gray in white matrix.

Fig. 3. Deformation bands may be seen clearly in the slip line pattern.

Fig. 4. a) Polygonization structure of very pure aluminum that was shaped about 40% at a final rolling temperature of 420°C, b) polygonization structure of very pure aluminum that was shaped 30% at a final rolling temperature of 480°C, c) recrystallization begins on the macroscopic structural faults, a rolled out cast pore is shown in the figure.

If very pure aluminum is shaped at higher temperatures, these deformation bands polygonalize (Fig. 4a and b). The diameter of the small polygonal blocks is in a specific ratio to the flow voltage [3]. Macroscopic structural faults can be the starting point where recrystallization starts. Figure 4c illustrates this on rolled out cast pores.

Grain boundaries can be recognized very clearly by setting up a slip line pattern (Fig. 5). In this way small orientation differences can be proven in the vicinity of the grain broundary. Furthermore, with this technique rough textures can be distinguished. By combining information from the form of small etching grooves and the slip line pattern in combination with the color of the structures that are seen in polarized light the content of the texture components can be determined.

Fig. 5. The grain construction can be shown by the slip line pattern. The grain boundaries are shown very clearly.

Summary

The metallography process that is described enables the structure of aluminum to be developed in a short time in such a way that the following information is made possible:

a) the presence of two phases, their configurations and distribution,

b) the density and distribution of dislocations on the basis of etched figures,

c) the orientation of the crystals by means of etched figures, slip line patterns and color effects in polarized light,

d) recovery and recrystallization.

In addition, it makes an exact determination of the grain size possible.

In addition, the method is also suitable for AlMg3 and AlMgSi specimens as well as for Al 99.99 and Al 99.7.

Bibliography

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